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Standard Reference Materials:

Transmission Filters With Measured Optical Density at 1064 nm Wavelength—SRMs 2046, 2047, 2048, 2049, 2050, and 2051

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Alan L. Migdall and Raju U. Datla

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FOREWORD

Standard Reference Materials (SRMs) are certified reference materials (CRMs) issued by NIST that are well-characterized using state-of-the-art measurement methods and/or techniques for chemical composition and physical properties. They are used to ensure the accuracy and compatibility of measurement results in many diverse fields of science, industry, and technology both within the United States and throughout the world. For many of the nation's scientists and technologists, it is therefore of more than a passing interest to know the details of the philosophy and procedures used at NIST to use, produce, and certify SRMs. The NIST Special Publication Series is a series of publications used for this purpose and a list of these can be assessed through the Internet, http://ts.nist.gov/srm.

This 260 publication is dedicated to the dissemination of information on the value assignment and certification of SRMs 2046-2051, Transmission Filters with Measured Optical Density at 1065 nm Wavelength. This publication explains the theory which serves as the basis for the preparation, measurement values and uncertainties, certification, and use of this series of SRMs. In general, much more detail will be found in this publication than is generally allowed or desired in scientific journal articles. This publication should provide sufficient additional information so these SRMs can be utilized in new applications in diverse fields not foreseen at the time the SRMs were originally issued.

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ABSTRACT

Standard Reference Materials (SRMs) 2046-2051 are transmission filters and may be used for calibrating transmittance measurements made using lasers or infrared spectrophotometers, for attenuating the optical power with an accurately known transmittance at a wavelength of 1064 nm, and for characterizing the nonlinearity of detection systems. The external transmittance (which is expressed as optical density in this publication) of these filters has been measured at a wavelength of 1064 nm using a Nd:YAG laser and silicon diode detectors. The uncertainty associated with the measured optical density of each filter has been individually determined. The filters are made of colored glass with uncoated, polished surfaces. The filter plates are 51 mm x 51 mm, with thicknesses ranging from 1 mm to 6.4 mm which correspond to nominal optical densities from 1 to 6.

Keywords: Attenuation; infrared filter; Nd:YAG laser; optical density; photodiode detector; transmittance; uncertainty.

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1. Introduction

For over 20 years, NIST has developed instrumentation and produced standard reference materials for spectral transmittance measurements at ultraviolet, visible, and near infrared wavelengths [1,2]. High-accuracy spectrophotometers are commonly used for these measurements [3,4]. Recent advances in stable laser sources and sensitive linear photodiode detectors have allowed accurate measurements of infrared spectral transmittance for very low transmittance materials [5-8]. Both direct and heterodyne detection methods have been employed for measuring transmittance at wavelengths of 633 nm (He-Ne line), 1064 nm (Nd:YAG line), as well as 10.2 μm and 10.6 μm (CO_2 lines), for up to 10 decades of attenuation, as reviewed in the work of Gentile et al. [9]. These developments allow NIST to provide calibration services and calibrated neutral density filters over a wide range of attenuation.

Standard Reference Materials (SRMs) 2046 (nominal OD 1, thickness 1.0 mm), 2047 (nominal OD 2, thickness 2.2 mm), 2048 (nominal OD 3, thickness 3.2 mm), 2049 (nominal OD 4, thickness 4.2 mm), 2050 (nominal OD 5, thickness 5.4 mm), and 2051 (nominal OD 6, thickness 6.4 mm) are transmission filters and are primarily intended for use in calibrating transmittance measurements made with lasers or spectrophotometers, accurately attenuating the optical power, and characterizing detector nonlinearity. The optical density (OD) is defined as $-\log_{10}T$, where T is the external transmittance. The filters in this series are made of colored glass with polished (uncoated) surfaces. The lateral dimensions are 51 mm x 51 mm. The OD of these filters has been determined at a wavelength of 1064 nm. This special publication discusses in detail the measurement theory and instrumentation, material preparation, and uncertainty determination for the certification of these standard reference materials.

2. Material Preparation and Instructions for Use

The transmission filters are fabricated and polished by Laser Optics, Inc.,* using NG-9 ionically colored glass manufactured by Schott of Mainz, Germany [10, 11]. The glass filters are uncoated and optically smooth. Each surface is flat to within one tenth of the wavelength of a He-Ne laser (633 nm) and the wedge angle is less than 5 μrad (1 arc s). The filter plates are 51 mm x 51 mm, with thicknesses varying from 1 mm to 6.4 mm for different optical densities.

Each transmission filter is stored in a special container to minimize the contamination of filter surfaces. The hard-foam insert supports the filter by its edges and prevents any contact between the middle portion of the filter surface and the walls of the container. The filter may be held by the edges with soft plastic gloves or optical lens tissue. A metallic mount may be used to hold the edge of the filter (within 7 mm from the edge). The central portion of the filter should never be touched by fingers or any hard objects. Dust may be removed by blowing with clean, dry air.

Only the central 20 mm x 20 mm region should be used. It is desirable to average the measured transmittance of the filter at several positions around the center. The laser beam should be perpendicular (within 2° or 0.035 rad) to the filter surface to avoid translating the beam and increasing the path length within the filter. The laser power (flux) on the filter should not exceed 20 mW (300 mW/cm^2) to avoid excessive heating. Furthermore, the filter temperature should be between 21 °C and 27 °C during measurements.

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* The use of trade name or company name is for identification only and does not imply endorsement by the National Institute of Standards and Technology, nor does it imply that the material or product identified are necessarily the best available for the purpose.

3. Theory of Transmittance and Optical Density

The refractive index, n, of the glass material used for these SRMs is about 1.5 at visible and near-infrared wavelengths. At normal incidence, the reflectivity at the air-filter interface is $\rho = (n-1)^2/(n+1)^2 \approx 0.04$. The filters attenuate radiation mainly by absorption. The external transmittance T is

$$T = (1-\rho)^2 e^{-ad} \tag{1}$$

for normal incidence, where a is the absorption coefficient and d is the thickness of the filter. Multiple reflections between the two surfaces of the filter have negligible effect on the transmittance if $d > 2$ mm because of the strong absorption inside the filter and the weak reflection at the surfaces.[2] This greatly reduces the difficulty of measuring the external transmittance with a high accuracy. For filters with 10 % transmittance ($d \approx 1$ mm), however, interference between multiple reflections can cause a large uncertainty and eq (1) needs to be modified to include interference effects.[12,13] The uncertainty caused by neglecting multiple reflections is evaluated in section 5.3.

The optical density, defined by OD = $-\log_{10} T$, can be calculated from eq (1), viz.

$$\mathrm{OD} = ad \log_{10} e - 2\log_{10}(1-\rho) \tag{2}$$

The change in the optical density is proportional to the change in the thickness or the absorption coefficient. The second term in eq (2) is about 0.035. The room-temperature absorption coefficient for the glass materials used for these SRMs is about 2.16 mm^{-1} at 1064 nm. Therefore, filters of 1 mm thickness exhibit an OD near 1, filters of 2 mm thickness exhibit an OD near 2, and so on. An absolute difference of 0.001 in OD corresponds to a relative difference $\Delta T/T$ of 0.23 % in transmittance.

4. Measurement Techniques and Instrumentation

The measurement setup is shown in figure 1. The optical source is a continuous-wave (CW), arc lamp pumped, Nd:YAG laser with an output power of 3 W at 1064 nm. The laser beam is directed through a collimator (which reduces the beam diameter to about 1 mm), a polarizer and a power stabilizer, a lens, and then to a light-tight enclosure. A wedged quartz beamsplitter is placed inside the enclosure to produce two reflected beams (each with an optical power ≈4 % of the incident beam). The beam reflected from the first surface of the wedge goes to the stabilizer's feedback detector (FD) to maintain a constant optical power. The power is stabilized to better than 0.3 % rms fluctuation over several hours of operation. The beam reflected from the second surface of the wedge is sent to a monitor detector (MD) to normalize the input power, which further reduces the effect of power fluctuation. The transmitted beam passes through a shutter and the sample (or a reference), and is then reflected by a mirror to the signal detector (SD). Attenuating filters reduce the laser power so that the detectors are operated in their linear ranges. A baffle located at the middle of the enclosure prevents stray laser light from reaching the signal detector. A detector enclosure made of anodized aluminum is required for measuring filters with OD > 9. Transmittance measurements for OD ≈ 10 are discussed in a separate publication [14].

The signal detector is a Hamamatsu S1337 series silicon photodiode, with an area of 10 mm x 10 mm. The detector responsivity at 1064 nm is approximately 0.15 A/W. A built-in trans-impedance amplifier is used with linearized gain factors from 2 kΩ to 100 GΩ, yielding an overall responsivity of 300 V/W to 15 GV/W. A six-and-half digit voltmeter measures the dc voltage from the amplifier. The dynamic range and linearity of the detector/amplifier electronics were discussed in [5] and [6]. Another voltmeter is used to simultaneously measure the output voltage from the monitor detector/amplifier.

The filter is mounted on a copper holder with a thermistor placed on the holder to monitor the filter temperature during the measurement. The temperature dependence of the optical density is discussed in section 5.1. Two computer-controlled motors move the filter holder horizontally and vertically so that the laser beam can be positioned at a desired location of a sample or a reference. The reference is left blank for measuring filters of OD 1 to 4, and an OD 3 filter is used as the reference for measuring filters of OD 5 and 6. An automatic data acquisition program controls the motion of the shutter and the motors, takes readings from the voltmeters, and calculates the transmittance (and optical density) for each measurement.

Six measured values are required to determine the relative transmittance of the sample filter at a single position. The working equation is

$$T_{relative} = \frac{[(V_{s1} - V_{s0}) / V_m]_{sample}}{[(V_{s1} - V_{s0}) / V_m]_{reference}} \tag{3}$$

where V is the output voltage, subscripts s and m indicate signal detector and monitor detector, respectively, and subscripts 0 and 1 indicate shutter closed and open, respectively. The signal when the shutter is closed (V_{s0}) is subtracted from the output signal (V_{s1}) to eliminate background. The sample transmittance is the same as the relative transmittance for filters of OD 1 to 4 since the reference is blank.

The gain setting cannot be changed during each measurement. The signal-to-noise ratio is lower with higher OD filters since the resolution of the voltmeter is 1 µV. Therefore, a reference substitution method is used to increase the dynamic range of the measurement. Filters of OD ≈ 5 and 6 were measured relative to a reference filter of OD ≈ 3. The transmittance of the reference filter was measured at a fixed position with a lower gain setting. The transmittance of the high OD filter relative to that of the reference filter was measured with a higher gain setting. The transmittance of OD 5 or 6 filters is calculated from

$$T_{sample} = T_{relative} \times T_{reference} \tag{4}$$

where $T_{reference}$ is the transmittance of the reference filter measured at a single fixed position. Eckerle et al. [2] used this "step-down" method for measuring spectral transmittance down to OD = 4.

The linearity of the detector was tested by varying the power levels on the detector using attenuation filters. The amplifier gain was optimized to yield the best signal-to-noise ratio without saturating the dc voltmeter. The optical density of an OD ≈ 4 filter measured with different incident laser powers is shown in figure 2. It can be seen that the detector is linear at optical powers less than 1 mW. At an input optical power of 1 nW, the power reaching the detector was ≈10^{-13} W when the beam passes through the OD 4 filter. The low signal-to-noise ratio resulted in a large uncertainty for the OD measurement at the low power end.

The output voltages from the monitor detector/amplifier and the signal detector/amplifier during typical measurements are shown in figure 3 to illustrate the power stability and the significance of the monitor detector. Only the output when the laser beam passes through the reference (blank) is shown. The background voltage of ≈40 μV is negligible compared to the signal voltage. The power stability varies from measurement to measurement, with figures 3a and 3b representing the best and worst cases, respectively. The relative standard deviations for both V_s and V_m are less than 0.04 % in figure 3a. Due to the high stability, the effect of normalization by the monitor detector, V_m in eq (3), is insignificant.

The normalization by V_m becomes important when the laser power variation is relatively large. In figure 3b, the relative standard deviations of the output signals are ≈0.4 %, i.e., about an order of magnitude greater than those in figure 3a. The outputs from MD and SD exhibited similar trends in figure 3b. In this case, the optical density of the sample filter calculated with and without normalization by V_m is compared in figure 4. The standard deviation in OD is significantly reduced when the laser power is normalized by the monitor detector output.

5. Effects of Temperature, Position, Interference, and Wavelength

The laser beam incident on the filter was perpendicular to the surface (angle of incidence < 2 °). This was checked by observing the reflected beam spot using an infrared sensing card. The slight translation of the beam through the filter (maximum 0.12 mm) has little effect on the measurement because of the high degree of spatial uniformity of the detector. The beam diameter at the filter was ≈3 mm full width at half maximum and the beam divergence is less than 2 mrad. The detector was tilted about 1° so that the beam was not reflected back on to the filter. The temperature and relative humidity of the laboratory were between 22 °C and 24 °C and between 40 % and 60 %, respectively, during the calibration measurements. Because there are no water absorption lines near 1064 nm and the refractive index of air is insensitive to humidity, the humidity level has negligible effect on the measurements.

5.1 Temperature Effect

The temperature of the filter was varied to investigate the effect on the optical density. The central air conditioner in the building was used to vary the temperature in the laboratory from 23 °C to 27 °C. The filter temperature was assumed to be the same as that of the copper holder. The filter temperature was about 1 °C higher than room temperature due to the heating of the positioning motors. To reduce the filter temperature below 24 °C, cold N_2 gas from a liquid-nitrogen tank was passed through the enclosure. The enclosure was then closed, and the optical density and filter temperature were monitored. The change of the filter temperature (1 °C to 2 °C per hour) was slow enough for a correlation between the OD and temperature to be obtained.

The measured OD versus temperature for two filters are shown in figure 5. The OD increases linearly with the filter temperature. The slight deviation at the low-temperature end is not surprising since the

temperatures of the filter and the holder may not have equilibrated for the first few data points (each measurement takes about 100 s). A linear fit shows that the OD increases 0.00052 /°C for the OD 2 filter and 0.0008 /°C for the OD 3 filter. This implies that the change in OD is caused by a change in the absorption coefficient of the glass material. Because the thermal expansion coefficient of the material is $\approx 6.3 \times 10^{-6}$ /°C [10], the change in thickness is negligibly small. For the OD 3 filter, the correlation between OD and temperature was also obtained as the temperature decreased with time (by reducing the laboratory temperature). The results, shown by the circles in figure 5b, agree well with those when the temperature is increased with time.

By substituting the measured OD and thickness values into eq (2), we found the absorption coefficient of the glass material to be ≈ 2.16 mm^{-1} at 25 °C with a temperature coefficient of ≈ 0.026 %/°C at a wavelength of 1064 nm. Therefore, a correction in the measured OD may be necessary for measurements at temperatures different from that indicated in the certificate. The correction depends on the nominal OD values. In the temperature range between 21 °C and 27 °C, the change in OD per °C temperature change for SRMs 2046, 2047, 2048, 2049, 2050, and 2051 is 0.00026, 0.00054, 0.00078, 0.00103, 0.00133, and 0.00157, respectively.

5.2 Spatial Nonuniformity

The spatial variation of OD depends on the filter. Measurements were performed either at 9 positions in a 3 x 3 matrix with 10 mm spacing or at 25 positions in a 5 x 5 matrix with a 5 mm spacing around the center of the filter. These measurements were repeated at least once for all positions. The measured optical densities of two filters at 25 positions are shown in figure 6. The OD 1 filter possesses a much larger spatial deviation in optical density than the OD 6 filter. Because the filter surfaces are extremely flat and parallel, the spatial variation is attributed to the inhomogeneity of the material with the exception for

OD 1 filters, where interference between multiple reflections may affect the spatial uniformity. Measurement using a micrometer (resolution of 2.5 μm) could not detect any variations in the filter thickness. The spatial nonuniformity is the major source of uncertainty for most filters.

5.3 Interference Effects

The transmittance of a plate with two parallel, optically smooth surfaces for completely coherent radiation is [12]

$$T = \frac{(1-\rho)^2 \tau}{1+\rho^2\tau^2 - 2\rho\tau\cos(2\pi\xi)} \tag{5}$$

where $\tau = e^{-ad}$ is the internal transmittance and ξ is a parameter defined as $\xi = 2nd / \lambda$. The transmittance oscillates as ξ varies. The amplitude of oscillation is estimated to be 0.8 % for OD 1 filters and 0.08 % for OD 2 filters. For OD 1 filters ($d \approx 1.044$ mm), a variation of either 0.18 μm in d or 0.17 nm in λ or 0.016 % in n would change the transmittance from a maximum to a minimum. The large spatial variation for OD 1 filters is caused by interference effects since the thickness variation is of the order of 0.2 μm. The spectral linewidth Δv of the laser is between 1 cm^{-1} and 5 cm^{-1} [15, 16]. Hence, the laser radiation is not completely coherent. Using the expression for partially coherent radiation given by Zhang [13], the transmittance can be calculated from

$$\begin{aligned} T = \frac{(1-\rho)^2 \tau}{1-\rho^2\tau^2}\Big[&1+2\rho\tau\cos(2\pi\xi)\ \mathrm{sinc}(2\pi nd\Delta v) \\ &+ 2\rho^2\tau^2\cos(4\pi\xi)\ \mathrm{sinc}(4\pi nd\Delta v) + \mathrm{H.O.T.}\Big] \end{aligned} \tag{6}$$

where the function sinc(x) = sin(x)/x, Δv is the laser spectral width, and H.O.T. indicates higher-order terms. Because $\rho^2\tau^2 < 2\times10^{-5}$, eq (6) can be approximated as

$$T \approx (1-\rho)^2 \tau \left[1+2\rho\tau\cos(2\pi\xi)\ \mathrm{sinc}(2\pi nd\Delta v)\right] \tag{7}$$

Figure 7a shows the predicted transmittance of an OD 1 filter (using a = 2.16 mm^{-1} and d = 1.044 mm) for Δv = 0, 1 cm^{-1}, and for incoherent radiation. The relative amplitude of oscillation is $\Delta T / T \approx 2\rho\tau \mathrm{sinc}(2\pi n d \Delta v)$. Using Δv = 1 cm^{-1}, the standard uncertainty in OD caused by interference effects (σ_{interf}) is estimated to be 0.0016 for OD 1 filters, 0.0001 for OD 2 filters, and negligible for filters of OD ≥ 3. As shown in figure 7b, variations in the range of ±0.0025 OD were observed for an OD 1 filter after subtracting the effect of absorption coefficient change, when the filter temperature was varied from 21 °C to 27 °C. This could be caused by a slight temperature dependence of n. The OD variation is less than $2\sigma_{interf}$, indicating that the calculated σ_{interf} on the basis of Δv = cm^{-1} is still a conservative estimate.

5.4 Wavelength Dependence

A Fourier transform infrared (FT-IR) spectrometer (configured with a halogen source, a quartz beamsplitter and a DTGS pyroelectric detector) was used to determine the wavelength dependence of the transmittance. The spectral transmittance for two filters is shown in figure 8 for wavelengths from 800 nm to 1400 nm. The spectral resolution was 8 cm^{-1} (≈0.9 nm at 1064 nm). The beam exiting the interferometer was focused at the filter with a ≈8 mm diameter spot size and a maximum divergence angle of ≈7°. The optical power was attenuated to improve the radiometric accuracy at the expense of a reduction of the signal-to-noise ratio. No absorption lines were observed and the transmittance spectra are relatively flat near 1064 nm. The spectral width of the Nd:YAG laser is less than 0.5 nm at 1064 nm [15, 16]. Transmittance values obtained using the laser and the spectrometer agree at the 1 % level, which is within the overall uncertainty of the two different measurement techniques. Hence, these filters are appropriate for use in calibrating infrared spectrometers at 1064 nm wavelength.

6. Uncertainty Determination

The standard uncertainty for measurement repeatability at the same position on the filter is better than 0.00006 for OD 1 and 2 filters, 0.0002 for OD 3, 5 and 6 filters, and 0.00067 for OD 4 filters (because of a lower signal-to-noise ratio). Table 1 lists the mean OD values averaged over 9 or 25 positions, the average filter temperature during the measurement, and all uncertainty components for six typical filters. The thickness of each filter measured using a micrometer is also listed in Table 1, with an expanded uncertainty (2σ) of 2.5 μm.

The uncertainty given in Table 1 associated with the nonuniformity ($\sigma_{spatial}$), which includes the repeatability component, is the standard deviation of the measurements at different positions of the filter. The standard uncertainty due to spatial nonuniformity ($\sigma_{spatial}$) was as large as 0.005 for some filters. Filters with $\sigma_{spatial} > 0.002$ were excluded from this certification. The reproducibility (σ_{reprod}) was determined from various sets of measurements of OD 1, 2, and 3 filters, performed on different dates when the samples had been unmounted and remounted. The standard uncertainty associated with the detector nonlinearity (σ_{nonlin}) was estimated to be 0.0002 for all filters. The standard uncertainty (σ_{temp}) caused by the temperature variation of ±0.5 °C was calculated from the temperature dependence of the absorption coefficient. The uncertainty due to the reference measurement (σ_{ref}) for filters with OD ≥ 5 is a combination of the nonlinearity, reproducibility, temperature effect, and the repeatability for measurements of the OD 3 reference filter at the fixed position. The combined standard uncertainty was calculated as a prediction interval, i.e.,

$$\sigma_{combined} = (\sigma_{spatial}^2 + \frac{1}{N}\sigma_{spatial}^2 + 2\sigma_{reprod}^2 + \sigma_{nonlin}^2 + \sigma_{temp}^2 + \sigma_{ref}^2 + \sigma_{interf}^2)^{1/2} \tag{8}$$

where N (9 or 25) is the number of measurement positions on the filter. The prediction interval, which includes twice the reproducibility variance

and (1 + 1/*N*) times the spatial-nonuniformity variance, gives users a basis to determine if a new measurement at any random position agrees with the certified value [17]. The expanded uncertainty (95 % confidence) is twice the combined standard uncertainty [18].

To determine the stability of the OD measurements, several filters were measured over a one-year period. The agreement in OD is within the expanded uncertainty of the measurements, indicating that these filters have good long-term stability.

7. Summary

We have developed and utilized an apparatus to characterize the optical density of colored glass filters (51 mm x 51 mm) at 1064 nm line of a Nd:YAG laser. The optical density of these filters ranges from OD 1 to OD 6 depending on filter thickness. The expanded uncertainty for the OD measurements is between 0.001 and 0.004 for all the certified filters, with inhomogeneity of the material being a major source of uncertainty for filters of OD > 1. Interference between multiple reflections causes a large uncertainty in the measurement of OD 1 filters. The effect of temperature on the OD was determined from 21 °C to 27 °C. The wavelength dependence and long-term stability were also investigated.

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Table 1 Thickness, optical density, filter temperature, and uncertainty components for six typical filters

Filter Number	10-01	22-01	32-01	42-01	54-01	64-01
Corresponding SRM Number	2046	2047	2048	2049	2050	2051
Thickness (mm)	1.044	2.177	3.157	4.194	5.410	6.391
Optical Density (OD)	1.0111	2.0785	2.9931	3.9679	5.1140	6.0325
Filter Temperature (°C)	24.7	25.4	23.8	24.1	25.1	25.3
Nonuniformity ($\sigma_{spatial}$)	0.00129	0.00065	0.00021	0.00072	0.00026	0.00023
Reproducibility (σ_{reprod})	0.00035	0.00035	0.00035	0.00035	0.00035	0.00035
Detector Nonlinearity (σ_{nonlin})	0.0002	0.0002	0.0002	0.0002	0.0002	0.0002
Temperature Variation (σ_{temp})	0.00007	0.00015	0.00022	0.00030	0.00038	0.00045
Reference (σ_{ref})	N/A	N/A	N/A	N/A	0.00050	0.00050
Interference Effects (σ_{interf})	0.0016	0.0001	N/A	N/A	N/A	N/A
Combined Standard Uncertainty ($\sigma_{combined}$)	0.00214	0.00087	0.00062	0.00097	0.00087	0.00089
Expanded Uncertainty	0.0043	0.0017	0.0012	0.0019	0.0017	0.0018

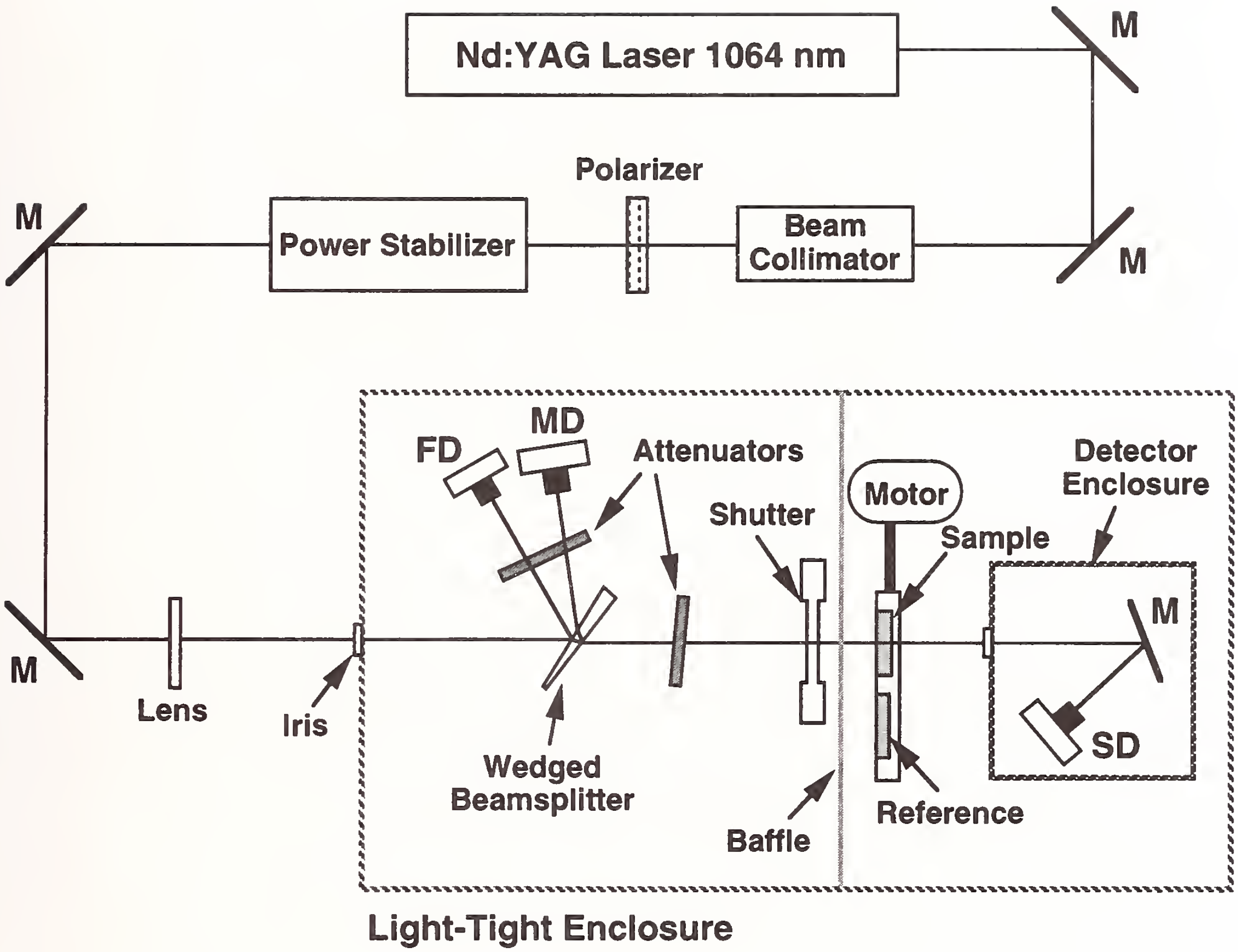


Figure 1. Schematic of the optical setup for transmittance measurements at 1064 nm wavelength, where M represents mirror; FD = feedback detector; MD = monitor detector; SD = signal detector.

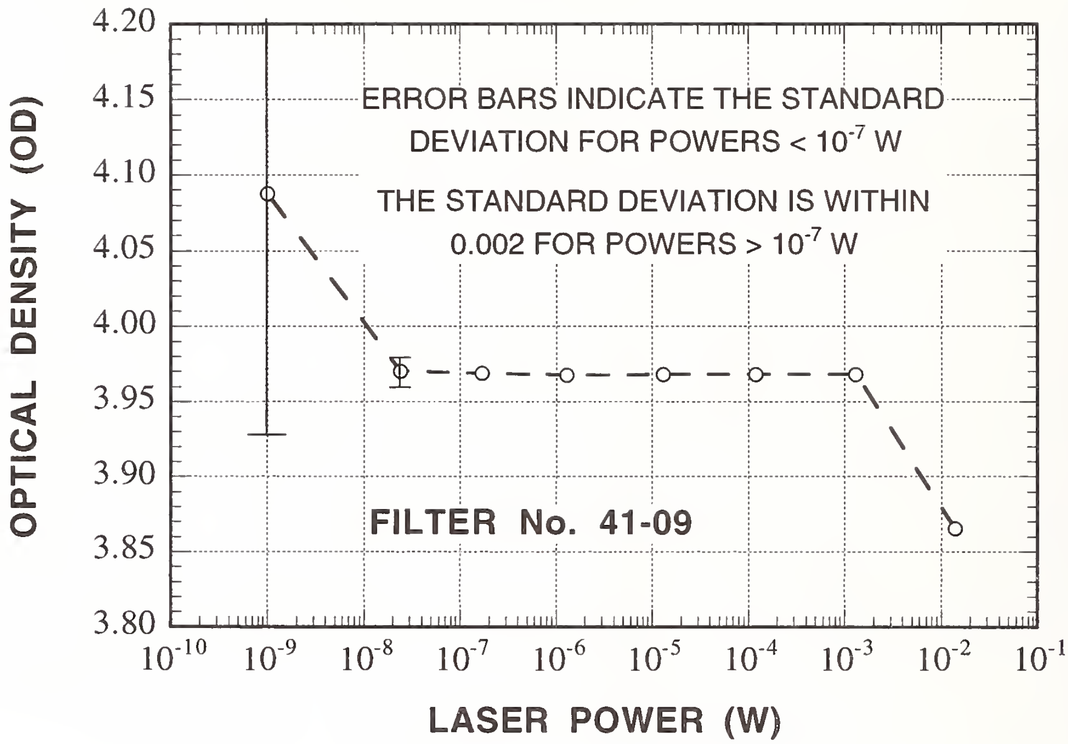
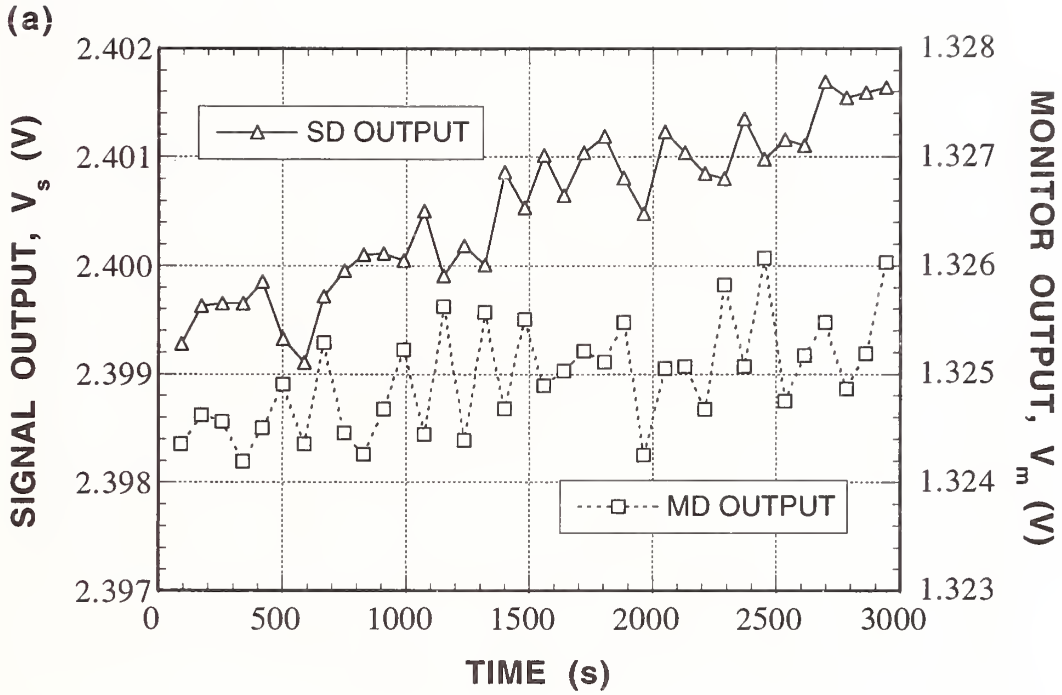


Figure 2. Optical density of a filter (nominal OD = 4) measured with different laser powers.



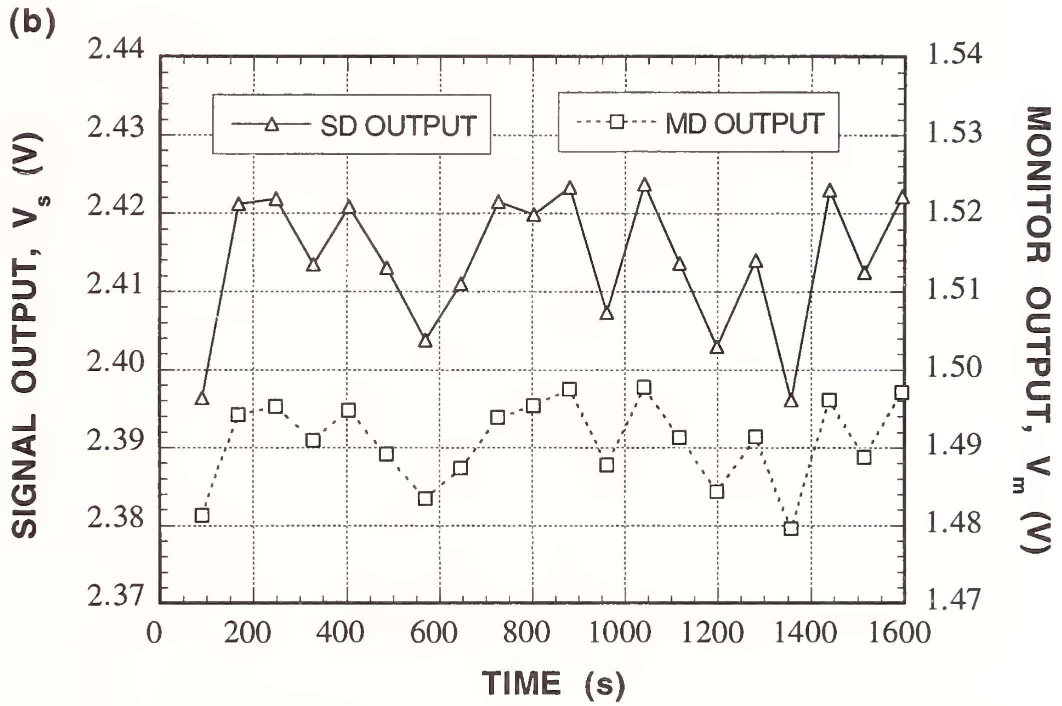


Figure 3. Outputs from the signal detector and the monitor detector during measurements when the laser beam passes through the reference. The laser power is more stable in a) than in b).

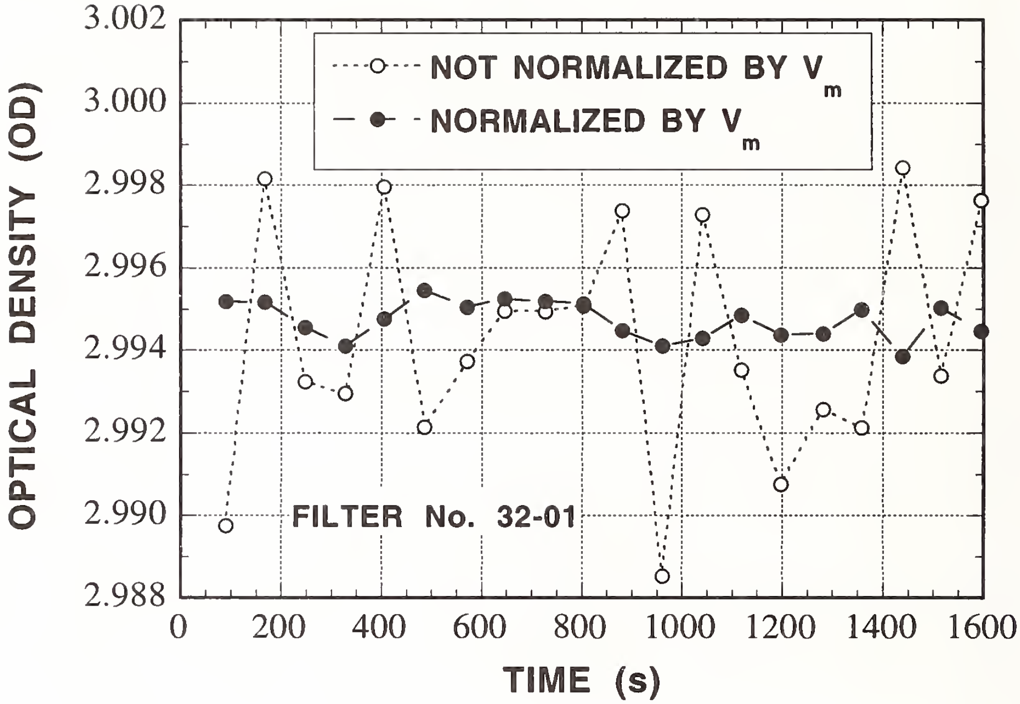
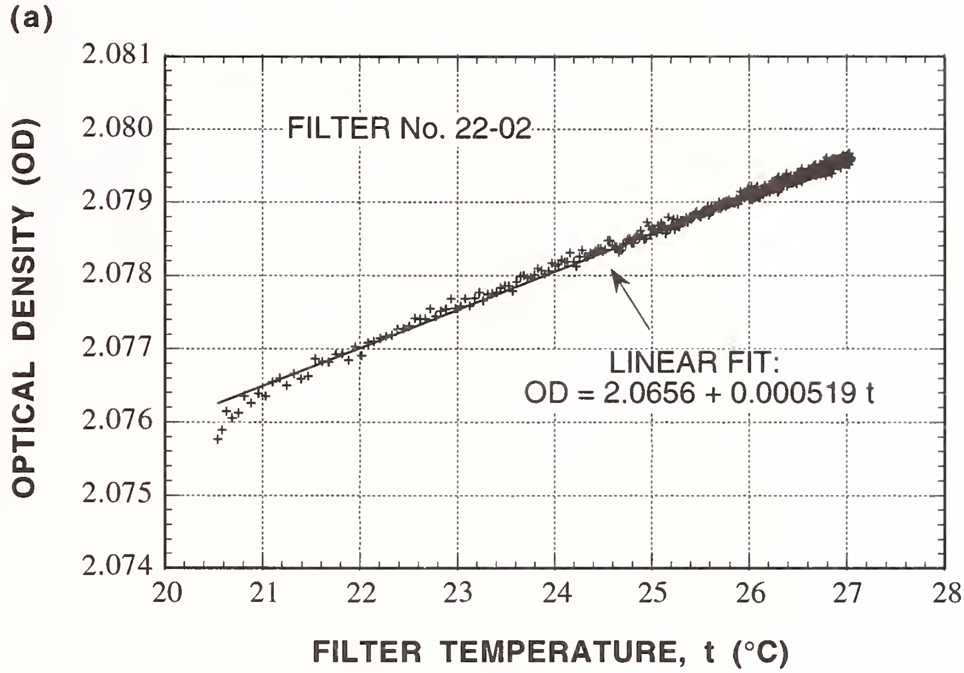


Figure 4. Comparison of the optical density calculated with and without normalization using the monitor detector output.



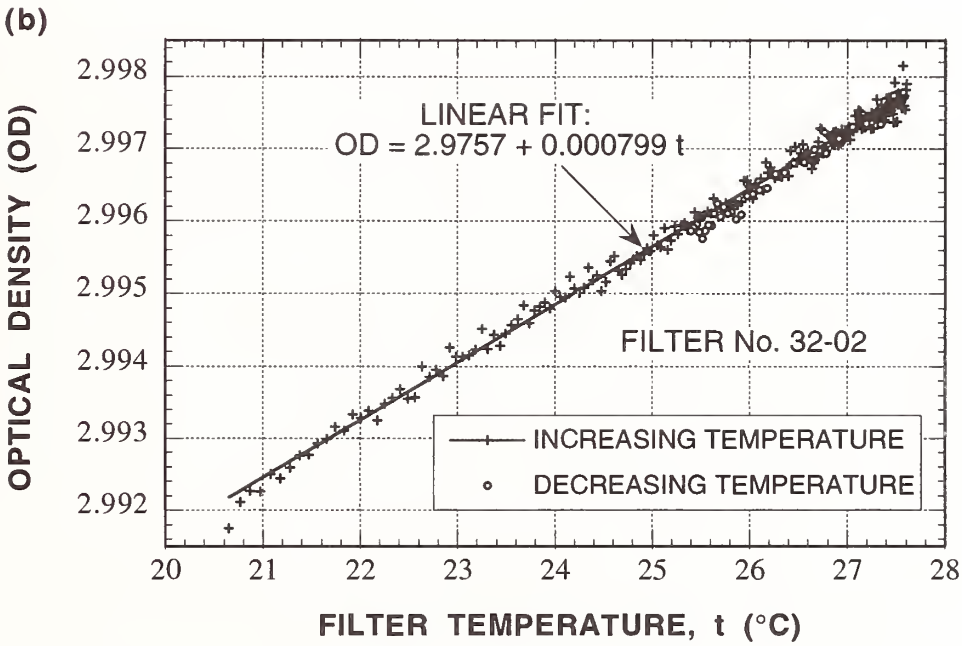
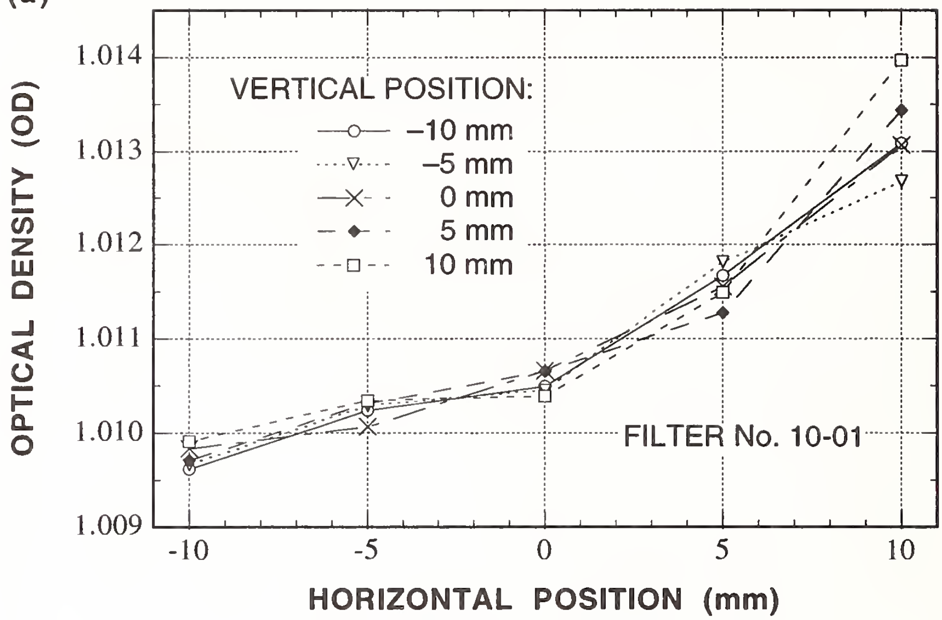


Figure 5. Optical density versus filter temperature for two filters: a) Nominal OD = 2; b) Nominal OD = 3.

(a)



(b)

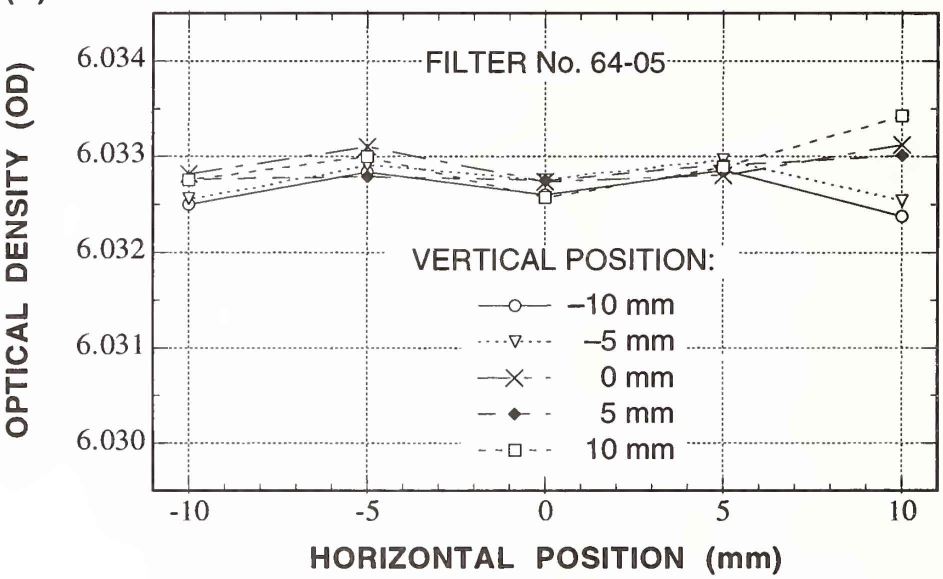


Figure 6. Spatial variation of the optical density for two filters, where the positions are measured from the center of the filter: a) Nominal OD = 1, relatively poor spatial uniformity; b) Nominal OD = 6, relatively good spatial uniformity.

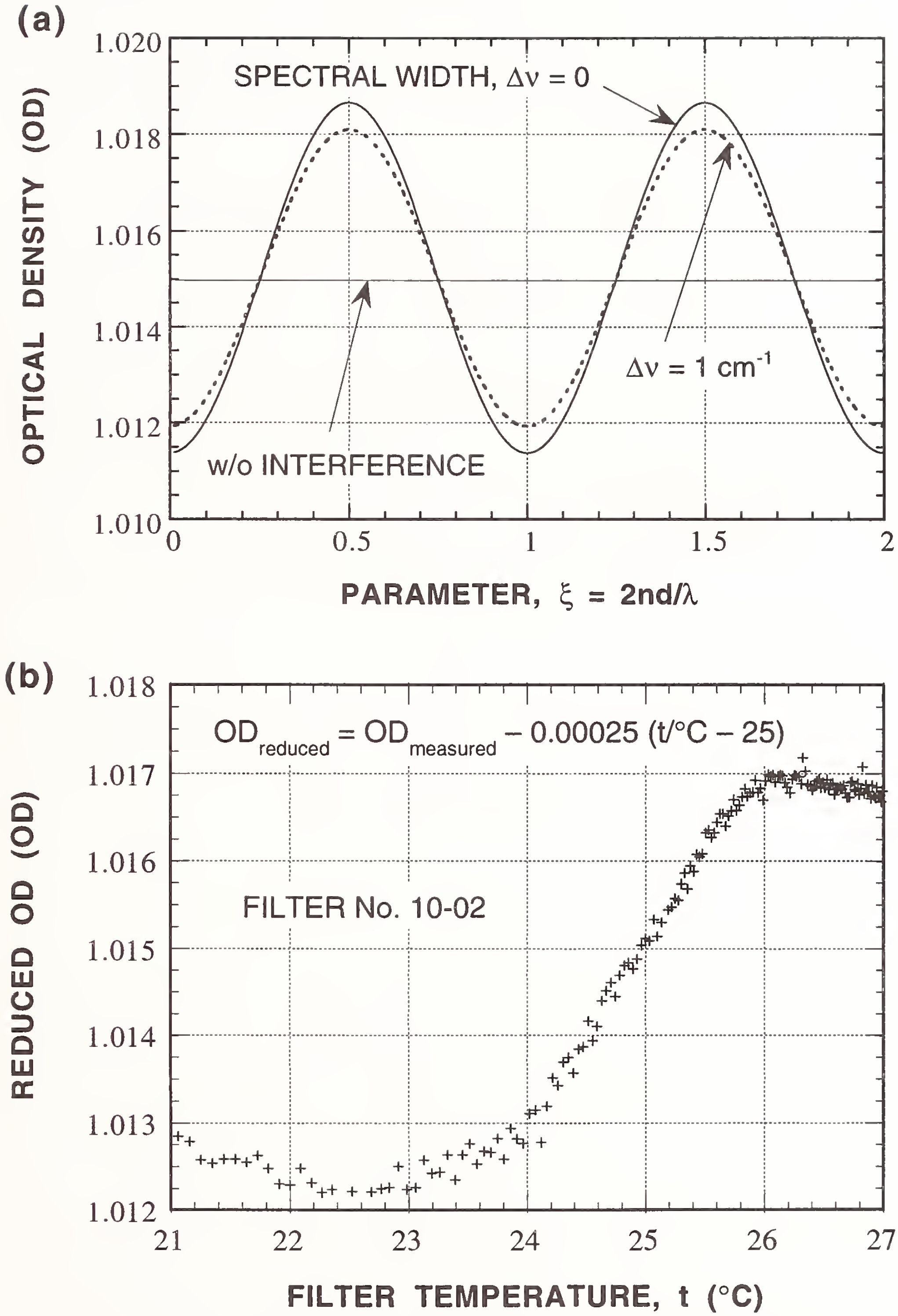


Figure 7. Interference effects on the transmittance: a) Predicted OD vs. $\xi = 2nd/\lambda$; b) Reduced optical density of an OD 1 filter as a function of temperature.

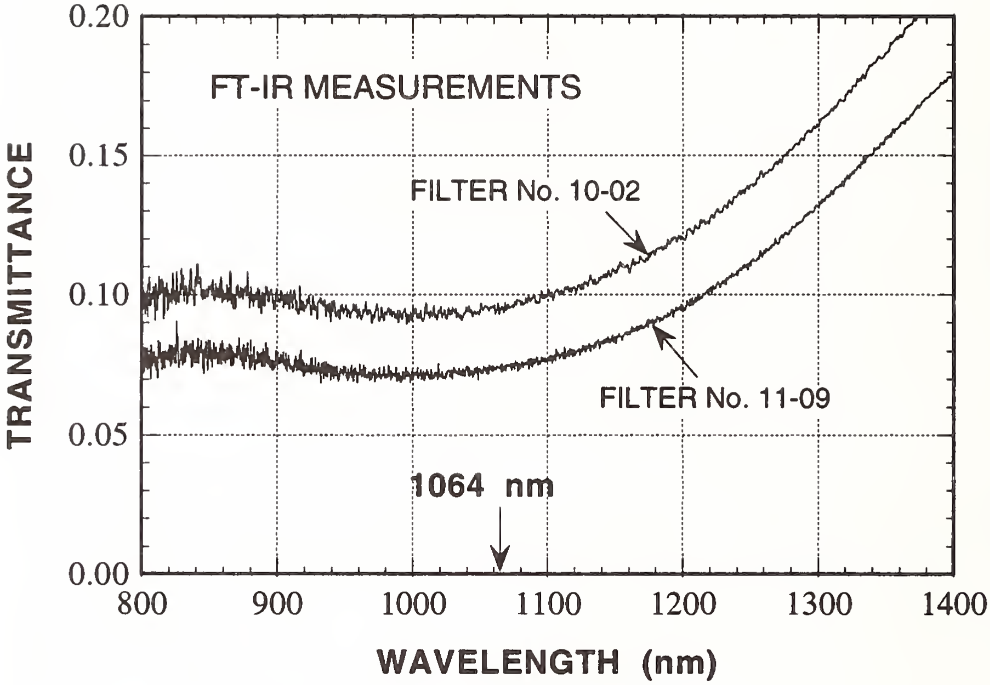


Figure 8. Transmittance in the region from 800 nm to 1400 nm of two filters measured using a Fourier transform infrared spectrometer.

National Institute of Standards & Technology

Certificate

Standard Reference Materials® 2046, 2047, 2048, 2049, 2050, 2051

Transmission Filters with Measured Optical Density at 1064 nm Wavelength

Serial No.

Standard Reference Materials (SRMs) 2046-2051 are a series of filters intended primarily for use in the calibration of transmittance measurements using lasers or infrared spectrophotometers; for attenuating the optical power with an accurately known transmittance at a wavelength of 1064 nm; and for characterizing the nonlinearity of detection systems. The filters are made of colored glass with uncoated, polished surfaces having dimensions of 51 mm x 51 mm. Each surface is flat to within one tenth of the helium-neon wavelength (633 nm), while the wedge angle is less than 5 μrad (1 arc s). The optical density (OD) of each filter has been determined at a wavelength of 1064 nm using a Nd-YAG laser and silicon diode detectors. The nominal optical density and the corresponding thickness of each SRM are given in Table 1.

Table 1. Nominal Optical Density with Corresponding Thickness

SRM Number	Nominal Optical Density	Thickness (mm)
2046	1	1.0
2047	2	2.2
2048	3	3.2
2049	4	4.2
2050	5	5.4
2051	6	6.4

Certified Values of the Optical Density: The certified optical density value and associated uncertainty for this filter are given in Table 2. The source and magnitude of each uncertainty component and the average temperature of the filter during the measurement are also listed in Table 2. The optical density is related to the transmittance, T, of the filter by OD = $-\log_{10} T$. An uncertainty of 0.001 in OD corresponds to a relative uncertainty $\Delta T/T$ of 0.23 % in transmittance. The certified OD value is the average of the measured values over the central 20 mm x 20 mm area of the filter. The certified values are for normal incidence (angle of incidence $\leq 2°$).

Expiration of Certification: The certification of the SRM is valid until **30 June 2003**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Instructions for Use). However, this certification is nullified if the SRM is damaged, contaminated, or modified.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.W.L. Thomas and J.C. Colbert.

Gaithersburg, MD 20899
Certificate Issue Date: 9 July 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

Overall Uncertainty Determination: The nonlinearity of the detector system was checked by measuring an OD 4 filter with different laser powers between 1 nW and 10 mW. The uncertainty associated with the detector nonlinearity was estimated to be 0.0002 for all filters. The reproducibility was determined from multiple measurements of OD 1, 2, and 3 filters performed on different dates. The interference effects added additional uncertainty for OD 1 and OD 2 filters. An uncertainty due to reference measurement is included in the combined standard uncertainty for OD 5 and OD 6 filters. The combined standard uncertainty was calculated as a prediction interval [1]. The uncertainty and its components for the given filter are listed in Table 3 [2].

Maintenance of SRM Certification: NIST will monitor these SRMs over the period of their certification. If substantive technical changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The development of the instrumentation and the measurements used to certify these SRMs were performed by Z.M. Zhang, T.R. Gentile, and A.L. Migdall of the NIST Optical Technology Division.

The overall direction and coordination of the technical measurements leading to certification were performed under the supervision of R.U. Datla of the NIST Optical Technology Division.

Statistical consultation was provided by M.C. Croarkin and S.B. Schiller of the NIST Statistical Engineering Division.

Source of Material: The filters were fabricated and polished by Laser Optics, Inc., using the NG-9 glass materials manufactured by Schott of Mainz, Germany.[1]

NOTICE AND WARNINGS TO USERS

Storage and Handling: The SRMs are stored in a wooden box, designed to minimize the contamination of the filter surfaces. The air gap in the box prevents any contact between the middle portion of the surfaces and the walls of the storage container. The filter may be held by the edges with soft plastic, powder-free gloves, or optical lens tissue. No filter mount is provided. A metallic mount may be used to hold the edge of the filter (within 7 mm from the edge). Care must be taken not to break the glass filter. The central portion of the filter should never be touched by fingers or any hard objects. Dust may be removed by blowing with clean, dry air.

Instructions for Use: Only the central 20 mm x 20 mm area of the filter should be used. It is desirable to integrate over several positions on the filter. The laser beam should be perpendicular (within 2° or 0.035 rad) to the filter surface since the filter attenuates the radiation through absorption. Because the optical density increases with increasing temperature at this wavelength, for filters with OD $\geq$ 3, a correction may be necessary if the filter temperature in the actual application differs from the value indicated in Table 2 (see the section entitled Temperature Dependence). The laser power on the filter should not exceed 20 mW (or 300 mW/cm^2) in order to avoid excessive heating.

CERTIFICATION ANALYSIS

Measurement Conditions: A continuous-wave (CW) Nd-YAG laser with an output wavelength of 1064 nm was used. The beam spot was approximately 3 mm in diameter (full width at half maximum). The laser beam incident on the filter was perpendicular to its surface. The temperature of the sample was monitored by a thermistor attached to the filter holder. The temperature and humidity in the measurement laboratory were between 22 °C and 24 °C and between 40 % and 60 %, respectively.

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[1] Certain commercial materials and equipment are identified in order to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are necessarily the best available for this purpose.

Determination of the Optical Density: A Nd-YAG laser and three silicon diode detectors were used to measure the normal transmittance of the filters at 1064 nm. The root-mean-square (rms) fluctuation of the laser power is less than 0.3 % over several hours of operation using a stabilizer. A wedged quartz beamsplitter was used. One of the reflected beams goes to a feedback detector that controls the laser stabilizer. The other beam reflected by the wedge is used to simultaneously monitor the laser power via the monitor detector. The transmitted beam passes through a shutter and the sample filter (or a reference), and then to the signal detector. The relative transmittance is

$$T_{relative} = \frac{[(V_{s1} - V_{s0}) / V_m]_{sample}}{[(V_{s1} - V_{s0}) / V_m]_{reference}} \qquad (1)$$

where V is the output voltage from the dc voltmeter, subscripts s and m indicate signal detector and monitor detector, respectively, and subscripts 0 and 1 indicate shutter closed and open, respectively. The detectors are placed inside a light-tight enclosure. The signal when the shutter is closed (V_{s0}) is subtracted from the output signal V_{s1} to eliminate background. For filters with OD ≤ 4, the reference is air (blank). Therefore, the sample transmittance is equal to the relative transmittance given in Equation 1. A reference filter with OD near 3 was used for filters with OD ≥ 5. The transmittance of the reference filter, $T_{reference}$, is measured at a fixed position. The transmittance of OD ≥ 5 filters is determined by

$$T_{sample} = T_{relative} \times T_{reference} \qquad (2)$$

The dynamic range and linearity of the detector and amplifier electronics are discussed in References [3] and [4]. The temperature of the filter is monitored by measuring the resistance of a thermistor on the filter holder during the data acquisition process. Detailed discussions of the theory and measurements are given in Reference [1].

Spatial Nonuniformity: The spatial variation in OD among different locations depends on the filter. Measurements were performed either on nine positions in a 3 x 3 matrix with 10 mm spacing or on 25 positions in a 5 x 5 matrix with a 5 mm spacing around the center of the filter. Because of the extremely flat and parallel surfaces of these filters, the spatial variation is attributed to the inhomogeneity of the material. For SRM 2046 OD 1 filters, interference between multiple reflections may also affect the spatial uniformity [3]. The measurement repeatability at the same position is better than 0.000 06 for OD 1 and 2 filters; 0.0002 for OD 3, 5, and 6 filters; and 0.000 67 for OD 4 filters (due to a lower signal-to-noise ratio). The uncertainty given in Table 3 associated with the nonuniformity ($\sigma_{spatial}$) is the standard deviation of the measurements on different positions of the filter, which includes the repeatability component.

Temperature Dependence: The OD increases slightly with temperature because of a change in the absorption coefficient of the material. The absorption coefficient of the glass material is 2.16 mm^{-1} at 25 °C with a temperature coefficient of 0.026 %/°C at a wavelength of 1064 nm [3]. Therefore, a correction in the measured OD may be necessary for measurements at temperatures different from that indicated in this certificate. The correction depends on the nominal OD values. Table 4 lists the change in OD per 1 °C temperature change at temperatures between 21 °C and 27 °C for different OD filters. The standard uncertainty resulting from the temperature variation of $\pm$ 0.5 °C is given in Table 3. The higher the OD of the filter is, the larger the standard uncertainty due to temperature variation.

REFERENCES

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[2] Zhang, Z.M., Gentile, T.R., Migdall, A.L., and Datla, R.U., *Optical Density Measurements for Infrared Filters at 1064 nm Wavelength*, NIST Special Publication 260-128, (1997).

[3] Eppeldauer, G. and Hardis, J.E., "Fourteen-Decade Photocurrent Measurements with Large-Area Silicon Photodiodes at Room Temperature," Applied Optics **30**, pp. 3091-3099, (1991).

[4] Migdall, A.L. and Winnewisser, C., "Linearity of a Silicon Photodiode at 30 MHz and Its Effect on Heterodyne Measurements," Journal of Research of the NIST **96**, pp. 143-146, (1991).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.

Table 2. Certified Optical Density at 1064 nm, SRM 2046
Serial No. 10-04 at Filter Temperature 24.8 °C[a]

Optical Density (OD):	1.0102
Expanded Uncertainty (2σ):	0.0035
[a]Nominal Thickness (in mm):	1.044

Table 3. Uncertainty Components, Serial No. 10-04

Spatial Nonuniformity ($\sigma_{spatial}$):	0.00045
Reproducibility (σ_{reprod}):	0.00035
Detector Nonlinearity (σ_{nonlin}):	0.0002
Temperature Variation (σ_{temp}):	0.00007
Reference (σ_{ref}):	0
Interference Effects (σ_{interf}):	0.0016
Combined Standard Uncertainty ($\sigma_{combined}$):	0.00175
Expanded Uncertainty ($2\ \sigma_{combined}$):	0.0035

Table 4. Change in OD for 1 °C Temperature Change

SRM No.	2046	2047	2048	2049	2050	2051
Nominal OD	1	2	3	4	5	6
ΔOD/°C	0.00026	0.00054	0.00078	0.00103	0.00133	0.00157

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